RESEARCH IN THE SYNTHESIS AND CHARACTERIZATION

OF MAGNETIC FERROFLUIDS

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ABSTRACT

This research studies the synthesis and characterization of strongly polarizable, extremely stable dispersions of magnetic media in a fluid carrier. Such fluids containing particles in the size range 50 to 300 angstroms are produced by wet grinding of ferrite powders in a ball mill. The fluids have a saturation magnetization on the order of 100 gauss at a viscosity of 10 centipoise. The viscosity may be reduced by a process of dialysis in which undersized material is removed. Electrodeposition of iron into mercury yields a magnetic colloid characterized by a particle size of 50 angstroms, a magnetic moment as high as 700 gauss, and a viscosity which is shear dependent and ranges in the thousands of centipoises. There is some evidence that bismuth additive greatly reduces the viscosity. A theoretical treatment recovers average size and standard deviation of particle size from magnetization curves.

NOTICE

This is the first of a series of quarterly reports under a contract with the National Aeronautics and Space Administration to provide research in the synthesis and characterization of magnetically polarizable colloidal fluids.

Major personnel who participated in the effort this quarter were Dr. J.W. Nestor, Dr. R.E. Rosensweig, Mr. N. Sheppard, and Mr. C. Panza.

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A. INTRODUCTION

This is the first of a series of quarterly reports under a contract with the National Aeronautics and Space Administration to provide research in the synthesis and characterization of ferrohydrodynamic fluids.

Ferrohydrodynamic fluids are strongly polarizable, extremely stable dispersions of magnetic media in a fluid carrier. In the presence of a non-uniform magnetic field these fluids experience an appreciable body force and hence give rise to a number of unique fluid mechanical flow phenomena. A basic fluid mechanical treatment has been given[1] for the flow of magnetic fluids and their interaction with thermal and magnetic fields. Another study[2] evaluated the feasibility of a new means of direct energy conversion that postulates the use of a magnetizable fluid whose magnetic moment is a function of temperature. Fluid rather than solid working substance was specified in the device to permit heat exchange operations that lead to a high predicted thermodynamic efficiency.

The key problem in the application of magnetic fluids at the present time is the development of satisfactory fluids. Ferromagnetism demands a domain or cooperative structure in matter that is not found in purely liquid systems. Accordingly, this research is directed to the production and study of stable suspensions of ferromagnetic particles in an appropriate liquid. Prior to embarking on the present program, Avco had demonstrated the ability to synthesize such suspensions by techniques which lead to the dispersion of colloidal size particles.

One means of preparing ferrofluid utilizes ball milling of solid charge (ferrite) and carrier fluid (kerosene) in the presence of a suitable surfactant. The resultant ferrofluid is electrically non-conducting and has a low thermal conductivity as well. Another category of ferrofluids of interest would be colloidal dispersions of magnetic metals in liquid metals which would have high electrical and thermal conductivities. Amalgams of this sort may be produced by electrolysis at a liquid mercury cathode to yield a suspension of submicron size insoluble magnetic particles.

The objective of this work is to synthesize and characterize experimentally and theoretically the physical, chemical, and magnetic properties of fluids having a strong magnetic response and to define the properties and discover the laws and relations which govern these ferrofluids. Special effort will be made to achieve high magnetic moment simultaneous with high fluidity. Ultimate limits to the magnetic strength of these fluids consistent with their stability will be studied theoretically and experimentally.

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1. Neuringer, J.L. and Rosensweig, R.E., Physics of Fluids, 1964, 7, p. 1927.

2. Resler, E.L., Jr., and Rosensweig, R.E., A.I.A.A. Journal, 1964, 2, p. 1418.

B. OVERALL PROGRESS

General

A laboratory devoted exclusively to these ferrofluid studies has been made available and a portion of this quarters effort devoted to design, construction, and installation of equipment. Production of both organic and metallic ferrofluid is underway and physical properties of some of the material was obtained. Analysis of the data has been carried out where useful for orientation of the program.

New Laboratory Equipment

Ball mills are used in these studies in the wet grinding of ferrite powders to reduce the particle size to the colloidal range. Accordingly, three U.S. Stoneware ball mills were installed which currently are used to rotate eight inch long stainless steel mill jars with baffles, each jar having a capacity of 1.63 gallons. An acoustic enclosure was built and installed to reduce noise to a comfortable level.

Raw ground ferrite material is centrifuged with a Lourdes LCA-1 centrifuge that provides 17,000 gees acceleration. Centrifuged liquid may be concentrated through a process of vacuum evaporation. For this purpose two Rinco rotating high vacuum-type evaporators were installed together with ancillary heating baths, cold traps, and vacuum pump. Each evaporator has a volume of 1000 milliliters. These evaporators eliminate bumping and attendant hot spots and permit evaporation to occur more rapidly or at lower temperatures. They operate on the principle of spreading a thin film over a large surface area and subjecting the film to a reduced pressure.

Two large electromagnets are now installed in the laboratory where they are operated from a 50 ampere well regulated power supply. One magnet is a Harvey-Wells with water cooled coils and four inch diameter pole pieces. The other was constructed in-house and has 2 3/4 inch pole pieces which are hollowed axially to accommodate removal of test specimens from a search coil in measurement of magnetization curves. Both magnets will produce fields in excess of ten kilogauss. Magnetization measurements are done in conjunction with a Leeds and Northrop rotating mirror galvonometer and capsules to accommodate fluid samples are one inch long by one-quarter inch diameter and one-half inch long by one-eighth inch diameter. The magnetization measurements are calibrated against standard pure iron and pure nickel cylindrical samples.

Magnetization measurements under low applied fields (< 100 oersted) will be done in a specially designed toroidal apparatus with uniform windings that may be filled to contain a fluid ring. Calibration against a standard is unnecessary due to the simple field geometry and there are no demagnitization effects. The apparatus is provided with water cooling to provide thermal control.

An electrolysis cell was constructed for the preparation of magnetic mercury colloid. The cell accommodates 50 to 150 milliliters of mercury in a flat pool which serves as a cathode for electrolysis from aqueous solution. An Armco iron anode having the same area is positioned above the mercury surface to produce a uniform current density. The mercury is agitated by a stirrer to promote formation of spherical particles and the cell is immersed in a constant temperature bath.

Viscosity determinations are done with capillary tube viscometers of the Ubbelohde type and these are immersed in another constant temperature bath. Determination of higher viscosities and characterization of non-Newtonian fluids is performed with a Brookfield RV viscometer. Special cylindrical spindles are on order that will permit shear rate to be known in terms of inverse seconds rather than arbitrary units of RPM of spindle rotation.

C. EXPERIMENTAL AND THEORETICAL RESULTS

1. Organic Ferrofluid

One batch representing the output of three mills has been completely processed including three weeks of grinding, centrifugation and vacuum evaporation. The standard charge to each mill was 1250 milliliters of kerosene, 300 grams of magnetite, and 150 milliliters of oleic acid. The final product consisted of 1200 milliliters of magnetic fluid having a magnetization of 18 gauss at 116 oersted, 40 at 650, 73 at 1900, 85 at 3950, 90.5 at 5300 and 93 at 6600 up to 10,400. Its density indicated a packing fraction of 3.3% and the viscosity was slightly less than 10 centipoise. This material is a standard solution that will be useful in further characterization and experimentation. Twice as much material, in addition, is in process.

2. Metallic Ferrofluid

Work has been initiated on the preparation and characterization of magnetic mercury amalgams. Data were obtained regarding the viscosity and concentration of the amalgams.

The results of various electrodeposition experiments are listed in Table I from which the following salient points may be noted. The material is more magnetic by a factor greater than six compared to any ferrite-kerosene colloid we have made. The amount of iron contained in the amalgam as determined from the Faraday equivalents to the total coulombs of charge that flowed is consistent with the amount calculated from the measured magnetic moment of the amalgam. However, the content of iron determined by density measurement using pycnometer technique leads to large overestimates at times. This indicates that simple additivity of volumes does not attain.

The magnetic mercury is separable into a magnetic portion and a non-magnetic portion. The magnetic portion is very viscous and usually displays a finite yield stress. Thus the free surface of a small pool of the material may be deformed after which the material fails to recover its shape. Measurements of the viscosity with a Brookfield viscometer show that apparent viscosity is a smoothly decreasing function of shear rate. Typical of the data are the following measurements obtained with ED3: μ = 90,000 centipoise at 2 RPM, μ = 40,000 at 4, μ = 17,000 at 10, and μ = 11,000 at 20 at room temperature using spindle number 5.

A bismuth amalgam was prepared by electrolysis from $Bi(NO_3)_3 \cdot 5H_2O$ in dilute nitric acid using a platinum anode and 100 grams of mercury, running one ampere for a half hour. A portion of the material was added to a portion of ED3 material and magnetically concentrated. The product was very magnetic, seemed to have a low viscosity and no longer displayed a yield stress as mentioned above. A sequence of experiments using ED4 material resulted in a product more viscous than the original. It is apparent that further study of the variables is needed.

TABLE I

ELECTRODEPOSITION OF IRON PARTICLES

Sample	Volume of Mercury	Electrolyte	Electrolyte Concentration (molal)	pH	Voltage	Current (amps)	Time (min)	(a) Amalgam Density (gm/cc)	Magnetic Moment at H=10,000 oe.	PERCENT IRON BY VOLUME CALCULATED RESULTS (d) Faraday	(e) Density	(f) Magnetic Moment
			MEASURED VARIABLES									
ED1	(b) 66.5 cc	$FeSO_4 \cdot 7H_2O$	1.6	--	3.2 v.	4	145			1.9%		
ED2 (c)	66.5	$FeSO_4 \cdot 7H_2O$	1.6	2	3.4	4	161	13.17		2.1%	6.5%	
ED3	133	$FeCl_2 \cdot 4H_2O$	2.2	2	2.2	2	50	12.59	500 gauss	2.7%	16.9%	2.3%
					4.0	5	150					
ED4	66.5	$FeSO_4 \cdot 7H_2O$	2.0	2	4.5	4	150		210	1.0%	1.8%	1.0%
ED5	66.5	$FeSO_4 \cdot 7H_2O$	1.6	1	3.0	3	360	13.38	705	3.5%	3.0%	3.3%
ED6	66.5	$FeSO_4 \cdot 7H_2O$	1.6	1	1.4	0.5	960	13.45		1.6%	1.8%	

(a) All samples were magnetically concentrated except ED1 and ED2
(b) Contained 5.5 grams of dissolved 20 mesh tin
(c) Subsequently electrolyzed with $SnCl_2 \cdot 2H_2O$ at pH1; 4 volts and 0.5 amp for 8 hours
(d) Assumes 100% cathodic efficiency
(e) Calculated from densities as $(\rho_{Hg} - \rho_{amalgam})/(\rho_{Hg} - \rho_{Fe})$
(f) Assumes magnetic moment of saturated iron of 21,580 gauss

3. Size Spectrum of Ferrite Dispersion

An electron micrograph of nickel zinc ferrite (Curie temperature 90-100°C) dispersed in kerosene and stabilized with oleic acid was examined to determine the distribution of particle sizes. The data were processed to yield $f_N(D)$, the number distribution function which gives the fraction of particles with diameters between D and D + dD per increment dD. Also obtained was $f_V(D)$, the distribution function for particle volumes.

$$\int_0^\infty f_N(D)\, dD = 1 \tag{1}$$

$$\int_0^\infty f_V(D)\, dD = 1 \tag{2}$$

The two distribution functions are related as follows, provided the particles are all similarly shaped.

$$f_V(D) = \frac{D^3 f_N(D)}{\int_0^\infty D^3 f_N(D)\, dD} \tag{3}$$

A simple exponential used to fit the data is of the following form:

$$f_N(D) = f_N(0)\, e^{-D/D^*} \tag{4}$$

where $f_N(D)$ and D^* are constants. From Eq. (1) it is required that $f_N(D) - 1/D^*$ so that

$$f_N(D) = \frac{1}{D^*}\, e^{-D/D^*} \tag{5}$$

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The data gave a value of $\overline{D}^* = 65$ angstroms. This is the same as the number average particle size $\overline{D}_N$ since

$$\overline{D}_N = \int_0^\infty D f_N(D) dD = \int_0^\infty \frac{D}{D^*} e^{-D/D^*} dD = D^*$$

(6)

An analytical expression for $f_V(D)$ follows from (3) and (5) as

$$f_V(D) = \frac{\frac{1}{D^*} \frac{D^3}{D^{*3}} e^{-D/D^*}}{\int_0^\infty x^3 e^{-x} dx} = \frac{1}{6 D^*} \left(\frac{D}{D^*}\right)^3 e^{-D/D^*}$$

(7)

This function passes through a maximum at $D = 3D^*$, hence on this basis the typical particle size appears to be larger than might be associated with a plot of $f_N(D)$. The volume averaged particle size $\overline{D}_V$ is given by

$$\overline{D}_V = \int_0^\infty D f_V(D) dD = \frac{D^*}{6} \int_0^\infty \left(\frac{D}{D^*}\right)^4 e^{-D/D^*} d\left(\frac{D}{D^*}\right) = 4 D^*$$

(8)

Thus, while $\overline{D}_N = 65$ angstroms the value of $\overline{D}_V = 260$ A$^\circ$ for the sample studied.

4. Particle Size and Distribution Related to Magnetization Curve

The particles in a ferrofluid as currently produced have a distribution of sizes. The following briefly describes a means for analyzing magnetization data to determine certain characteristics of the initially unknown distribution. The results of a determination are also given.

The Langevin function L(a) describes the net alignment averaged over a collection of non-interacting particles of uniform size in response to an applied field H when subject to thermal disorientation at temperature T.

$$L(a) = \coth a - \frac{1}{a} \qquad a = \frac{v M_s H}{kT}$$

(9)

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where v is the subdomain particle volume, M_S is the spontaneous magnetization of a domain, i.e., the magnetic moment per unit volume, and k is the Boltzmann constant. In low fields this becomes

$$\frac{M}{\epsilon M_S} = \frac{v M_S H}{3kT} \times \frac{1}{4\pi}$$

(10)

where M is the bulk magnetic moment per unit volume and ϵ the packing fraction of magnetic material. At the other extreme of high applied fields one has instead,

$$\frac{M}{\epsilon M_S} = 1 - \frac{kT}{v M_S H} \times 4\pi$$

(11)

For a ferrofluid containing a distribution of sizes described by a distribution function f(v) which specifies the volume of particles per volume of ferrofluid and per unit increment dv of particle size, the magnetization is given by the following integration over the distribution function as weighted by the Langevin function

$$\frac{M}{\epsilon M_S} = \int_0^\infty f(v) \left\{ \coth \frac{v M_S H}{4\pi kT} - \frac{kT \cdot 4\pi}{v M_S H} \right\} dv$$

(12)

Thus for high applied fields, using (11) and carrying out the integration one obtains

$$\frac{M}{\epsilon M_S} = 1 - \frac{1}{\bar{v}} \left(\frac{kT \cdot 4\pi}{M_S H} \right) = 1 - \frac{H_k}{H}$$

(13)

where $\bar{v} = \epsilon/N$ is the average volume of a particle and N is the total number of particles per unit volume. Thus if experimental data are plotted with $M/\epsilon M_s$ as ordinate versus $1/H$, the tangent to the curve at $1/H$ equal zero will intersect the $1/H$ axis and thus define a characteristic field H_h. The average particle volume $\bar{v}$ is then given by

$$\bar{v} = \frac{kT \cdot 4\pi}{H_h M_s} \qquad (14)$$

A corresponding treatment in the case of low fields using (10) and (4) gives

$$\frac{M}{\epsilon M_s} = \frac{M_s H N}{4\pi \cdot 3 \epsilon kT} \overline{v^2} = \frac{H}{H_\ell} \qquad (15)$$

where $\overline{v^2}$ is the mean square particle volume and the characteristic field H_ℓ is easily found from a graphical construction based on $M/\epsilon M_s$ plotted versus H. Accordingly, from (14), (15) and $\bar{v} = \epsilon/N$,

$$\overline{v^2} = 3\left(\frac{4\pi\, kT}{M_s}\right)^2 \frac{1}{H_h H_\ell} \qquad (16)$$

The statistical variance of volume is given by $\overline{v^2} - (\bar{v})^2$ and hence from (14) and (16) one obtains for the ratio of standard deviation to the mean,

$$\frac{\left[\overline{v^2} - (\bar{v})^2\right]^{1/2}}{\bar{v}} = \left(3\frac{H_h}{H_\ell} - 1\right)^{1/2} \qquad (17)$$

Mercury amalgam ED5 was analyzed according to the above principles with the result that $H_h = 345$ oersted corresponding to $d = 50.5$ A° in general agreement with electron microscopy results where amalgam was contacted with 30% hydrogen peroxide and examination made of oxidized particles which separated from the mercury. In addition the magnetization data defined a field $H_\ell = 400$ oersted corresponding to a ratio of standard deviation to mean volume from (17) of 1.3.

5. Dialysis

The ferrofluids as currently produced contain a distribution of particle sizes. In the case of the ferrite containing fluids the particles have absorbed on their surface a layer of surfactant which gives the dispersion stability against coagulation. The smallest particles will have an amount of adsorbed material which greatly exceeds in volume the volume of the particle itself. Accordingly, the viscosity of the bulk fluid will be increased by this immobilized material which may be crudely considered as additional solid material in the mixture. Thus anything that can be done to remove the undersize material from the fluid should have the effect of reducing the viscosity.

During this quarter some experiments in dialysis have been made to test the above hypothesis. Ferrofluid was placed in a specially designed cylinder between two millipore filters of 47 mm. diameter with 80% open area of 1000 A° closely sized holes. The cylinder is rotated in a container holding kerosene and a process of diffusion takes place in which small particles are transported out of the cylindrical cell at a faster rate than large particles due to the difference in diffusion rates. At the same time kerosene diffuses into the cell and dilutes the sample.

Ferrofluid which initially had a density of 0.858 (corresponding to a solids loading of 2.2%) and a viscosity of 1.62 centipoise had, after dialysis, a density of 0.824 and a viscosity of 1.82 centipoise. An additional aliquot of the initial material was then diluted very close to the same density, namely 0.825, and its viscosity determined to be 2.18 centipoise. Thus dialysis succeeded in reducing the viscosity of a rather dilute ferrofluid by 16.5%. It is speculated that the effect would be more pronounced at higher concentrations.

D. CURRENT PROBLEMS AND PROPOSED CORRECTIVE ACTION

A major problem in this investigation is to study means for reducing the
viscosity and increasing the magnetization of the ferrofluids. When using a
given magnetic material the magnetization may be increased by increasing the
solids loading and this will also invariably increase the colloid viscosity.
Theoretical predictions are helpful for determining the viscosity of a fluid
containing suspended particles. The earliest is that of Einstein[3] which was
derived by solving the flow field of pure strain perturbed by the presence of
a sphere. The result related mixture viscosity μ_s to solvent viscosity μ_0 and
solids fraction ϕ ,

$$\frac{\mu_s}{\mu_0} = 1 + 2.5\phi \tag{18}$$

This relationship is valid only for small concentrations such that the radius r
of particles is small compared to the mean distance of separation. For higher
concentrations one may consider the procedure of DeBruyn[4] and assume a
functional relationship of the type:

$$\frac{\mu_s}{\mu_0} = \frac{1}{1 + a\phi + b\phi^2} \tag{19}$$

For very small values of ϕ it is insisted that this equation reduce to Einstein's
which thus determines a = -2.5. At some concentration ϕ_c indicative of close
packing the fluid mixture becomes rigid and so the fluidity μ_0/μ_s goes to
zero. This determines the constant b as:

$$b = \frac{2.5\phi_c - 1}{\phi_c^2} \tag{20}$$

Hence the viscosity relationship (19) can be written as:

$$\frac{\mu_s - \mu_0}{\mu_s} = 2.5\phi - \frac{2.5\phi_c - 1}{\phi_c^2}\phi^2 \tag{21}$$

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3. Einstein, Albert, "Investigations on the Theory of the Brownian Movement",
    1956, p. 12 (New York: Dover Publications, Inc.)
4. See Kruyt, H.R., "Colloid Science" 1952, Vol. 1. p. 351 (New York: Elsevier
    Publishing Co.)

DeBruyn used the value $\phi_c = 0.74$ corresponding to hexagonal close-packed spheres.

For kerosene with 10% by volume of ferrite, Eq. (21) with $\phi = 0.10$ computes $\mu_s/\mu_o = 1.31$ whereas the measured value for a manganese zinc ferrite colloid prepared in our earlier work is the much higher value of 11.1 corresponding to kerosene viscosity of 1.6 centipoise and mixture viscosity of 17.8 centipoise. It is reasonable to expect that the adsorbed layer of oleic acid is effective in increasing the rigid volume of the suspended particles since the length of an oleic acid molecule has been determined to be 11.2 A°.[5] Uncoated spherical particles of radius r, when present in such quantity as to give a volume fraction ϵ, will, when coated with a uniform layer of thickness, δ, occupy a fractional volume in the fluid of:

$$\phi = \epsilon \left(1 + \frac{\delta}{r} \right)^3 \tag{22}$$

The foregoing equations may thus be combined to give the following:

$$\frac{\mu_s - \mu_o}{\epsilon \mu_s} = 2.5 \left(1 + \frac{\delta}{r} \right)^3 - \left(\frac{2.5\phi_c - 1}{\phi_c^2} \right) \left(1 + \frac{\delta}{r} \right)^6 \epsilon \tag{23}$$

A plot of measured values of $(\mu_s - \mu_o)/\epsilon \mu_s$ versus ϵ should yield a straight line and this is confirmed by our data. Furthermore, the value of δ/r determined from the slope agrees with that determined from the intercept, namely $\delta/r = 0.85$. Accordingly, one may calculate that if δ/r can be reduced to one-quarter of its present value it should be possible to obtain a fluid whose volumetric loading of magnetic material is the same as a present fluid, about 10%, but whose viscosity is only one-tenth of the present value; alternatively, at the same viscosity the concentration would be three to four times greater, i.e., 30-40 percent. Reduction in the ratio of δ to r might be done either by reducing δ or increasing r. Detailed considerations of stabilization indicate that reduction of δ is not a promising approach. However, the fluid now produced does contain a fraction of particles in the larger size range and hence it is desirable to attempt their separation or increase their formation relative to other sizes.

Accordingly, it is planned to study the nature of material generated in the grinding process as a function of time with the objective of seeking an optimum time to terminate the grinding. It is also planned to vary

5. Taylor, H.S. and Glasstone, S. (Eds.), "A Treatise on Physical Chemistry" 1951 Third Edition, Vol. II, p. 588 (New York: D. Van Nostrand Company, Inc.)

the nature of the surfactant. Further dialysis studies are planned, and fractional centrifugation will be considered as an alternate technique.

In the case of the mercury amalgams it is planned to vary particle size and study the effect on viscosity in a series of temperature aging runs. Based on the data of Luborsky[6] (see his Fig. 3) it appears that successive one day soaks at the indicated temperatures will give the corresponding particle sizes as shown in Table II.

TABLE II

AGING OF IRON-MERCURY COLLOID INITIAL DIAMETER = 50 A$^\circ$

DAY	TEMPERATURE	DIAMETER
1	140°C	100 A$^\circ$
2	180°C	200 A$^\circ$
3	200°C	300 A$^\circ$
4	220°C	400 A$^\circ$
5	250°C	600 A$^\circ$

It is also planned to more carefully study the effect of bismuth additive on the properties of the mercury colloid. Following this, a number of other metallic additives may be tried. For example, the following metals do not form compounds with iron and have the indicated solubilities in mercury at 25 $^\circ$C. They are lead (1.5 weight percent), cadmium (6 percent), indium (55 percent), and silver (.035 percent). The solubility of bismuth is 1.4 percent.

6. Luborsky, F.E., "The Kinetics of Growth of Spherical Iron Crystallites in Mercury", Jl. of Phys. Chem. 61, 1336 (1957).

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